

Performance Monitoring Protocol (QA/QC) for the Thermo Nicolet FTIR

1 Scope

This document addresses the performance monitoring (QA/QC) of the Thermo Nicolet Fourier Transform Infrared (FTIR) bench spectrometer and various accessories. This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives (chemistry) examinations performed at the Huntsville facility.

2 Principle

The Thermo Nicolet FTIR is a bench spectrometer that can be used by itself or in conjunction with an accessory. The accessories include an integrated iS50 Attenuated Total Reflectance (ATR) and an iD7 ATR. Definitions and guidelines for following this protocol are outlined in the “General Instrument Maintenance Protocol.”

3 Equipment/Materials/Reagents

- a. Thermo Nicolet iS50 or iS5 FTIR, and Omnic Software (or equivalent)
- b. ATR Accessories: integrated iS50 or iD7 (or equivalent)
- c. Polystyrene Standards - 1.5 mil film mounted on a card, or mounted in a metal frame

4 Standards and Controls

4.1 Performance Standards

The polystyrene films are used to assess operating performance, wavenumber assignment, and continued integrity of the system. The polystyrene standards used for this procedure require no preparation and do not expire. It is recommended by Thermo Nicolet that they are replaced if showing signs of wear or if results have drifted.

5 Sampling

Not applicable.

6 Procedures

6.1 Daily Checks

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

- a. Choose the appropriate bench and/or accessory.
- b. Load a method that is appropriate for the bench/accessory and mode of detection (ATR or %T) being used. Verify all parameters using the 'Instrumental Conditions' section of this protocol.
- c. Collect a background and sample spectrum of the polystyrene standard.
- d. Use 'Find Peaks' to label the major peak apexes. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the spectrum.
- e. Save the spectrum to the QA/QC polystyrene standards folder.
- f. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, align the bench/accessory and re-analyze the polystyrene. If the results are still poor or failing, contact the appropriate instrument support personnel.

6.2 As Needed Checks

The following steps are to be performed as needed based on system performance, and can be performed more frequently if desired. If a problem is indicated by the failure of the 'Daily Checks', these steps can help to identify the cause of an instrument error. Indicate completion in the appropriate log.

6.2.1 Interferogram Signal Evaluation

- a. Select the method (mode of detection) 'Bench %T' or equivalent.
- b. Clear the sample compartment of any material which would impede the IR beam.
- c. Monitor the interferogram signal under a gain setting of one (1.0).
- d. Record the peak-to-peak voltage of the interferogram (which is the sum of the absolute minimum and maximum peak values) in the appropriate log. This value reflects the amount of signal (in terms of voltage) being detected.

- e. If the signal value has dropped significantly, the beamsplitter can be automatically adjusted to improve the beam voltage throughput. Refer to the manufacturer's instrument manuals for further instructions or contact the appropriate instrument support personnel.

6.2.2 Bench Evaluation

- a. Initialize the appropriate system validation/qualification program (ex. Val-Q/ValPro) from within Omnic.
- b. Open the appropriate bench.csv file (if applicable).
- c. Start the validation. The iS50 will internally supply the polystyrene standards.
- d. Evaluate the validation report. It will specify if all tests pass or if any fail. If any fail, align the bench and repeat. When the results are acceptable, print the report. Printing the spectral data is optional.
- e. Save the spreadsheet data to the hard drive
- f. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, the appropriate instrument personnel will determine the corrective action to be taken.

7 Instrumental Conditions

Refer to the "General Instrument Maintenance Protocol" for procedures on minor deviations.

7.1 ATR Accessory

Mode:	Reflectance
Number of scans:	32
Resolution:	4
Scan range:	minimum 400 – maximum 4000 cm^{-1}

8 Decision Criteria

8.1 Polystyrene

The Polystyrene spectrum is acceptable if the following four peaks are within a $\pm 4 \text{ cm}^{-1}$ window of the expected wavenumber. If values lie outside the specified range, align the bench/accessory

and re-analyze the polystyrene. If the results are still poor or failing, contact the appropriate instrument support personnel. The following values have been provided by Nicolet:

<u>Expected</u>	<u>Acceptable Range</u>
3025 cm^{-1}	3021 to 3029 cm^{-1}
1601 cm^{-1}	1597 to 1605 cm^{-1}
1028 cm^{-1}	1024 to 1032 cm^{-1}
906 cm^{-1}	902 to 910 cm^{-1}

8.2 Validation Report

The Validation Report generated will indicate whether the obtained values lie within the ranges specified by the manufacturer and provide a 'pass' or 'fail' result. All tests should pass.

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel shall perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis.

13 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

“General Instrument Maintenance Protocol” (IOG 001) *Instrument Operations Group SOP Manual*.

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	10/04/18	New document that specifies instrument protocol for the Huntsville facility.

Approval

Redacted - Signatures on File

Scientific Analysis
Unit Chief

Date: 10/03/2018

TL Approval

Explosives (Chemistry)
Technical Leader

Date: 10/03/2018

QA Approval

Quality Manager

Date: 10/03/2018